Summary of the Discussion during the 1st Workshop for the High Resolution Powder Diffraction Beamline

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The summary below is based on the discussion with the user community at the end of the high-resolution workshop on 26th of November 2009. There were ~ 30 participants from different communities that will use the HRPD beamline.

Optics:

- The undulator optimized for high photon energies and the double Laue monochromator with fixed energy at 60 keV are constrained by
    1. The operation of two beamlines with one undulator,
    2. The space constraints in the optics hutch P02 and the maximal separation between the two beamline.
- Absorption edges close to the energy of 60 keV are Thulium at 59.3 keV and Ytterbium at 61.3 keV, respectively. Thus, there should be no fluorescence from typical absorber elements like Tungsten or Tantalum.
- Diamond crystals for the double Laue mono have arrived (with 1 and 40 A roughness, respectively) but are not of optimal thickness. However, the thickness of 400 vs. 345 μm (maximum flux at 165 and 345 μm) might only result in a 10% flux decrease for the HRPD beamline and should have no noticeable effect for the ECB. The group FS-BT is willing to polish diamond crystal to the optimal thickness if flux could be improved (only on 40 A rough crystal). Initial test will be conducted with the 400 μm thick crystal. With increased thickness heat load on the diamond crystal should be reevaluated.
- Time resolved measurements require as much flux as possible and hence an option to focus the beam might be useful. CRL lens changer has been proposed for a later stage of the beamline development. Such a device could be positioned in the optic hutch of P03 or the experimental station P02.1. Focusing would also satisfy the variable beam sizes requested for stress and strain measurements. Improvement of the resolution of powder diffraction experiments with the 2D detector by focusing on the area detector will be minor since the pixel size of the area detector (200 μm for PerkinElmer) is already very close to the beam size (500 (H) x 400 (V) μm^2). Refocusing of the beam would only make sense if pixel size is significantly reduced. Furthermore, focusing can result in poor count statistics since the beam size will approach the grain size of the sample. Hence the recommendation to make focusing optional and easy to remove. Overall, the focusing option is a low priority item.

Diffractometer

- Companies that are offering diffractometers with the required specs (load capacity and angular resolution) are RPI (ESRF and Soleil), Newport (Diamond) and Huber (ALBA).

- Analyzer crystals of Ge result in a factor 2 more flux on the detector but cause significant decrease in resolution and a broader shape of the powder diffraction peaks. Neither the Ge nor the Si analyzer crystals oxidize at the flux originating from the sample. Diamond analyzer crystals are not really an alternative because they are too expensive for the required size and would have to be installed in Laue geometry. Latter would result in the transmitted beam hitting the housing and cause additional noise (cross talking) in adjacent detectors.

2-D detector:

- Different fast 2D detectors are available: MAR555 (selenium direct x-ray conversion technology), PerkinElmer and Pixium (CsI bonded to amorphous silicon). The MAR555 has the smallest pixel size and Point Spread Function (PSF) and the highest dynamic range, but also significant readout problems (even though MAR has made significant progress in improving the detector performance). Nevertheless, the pixel size is less important for time resolved experiments and it will be better to use a detector with fast and reliable readout such as the PerkinElmer. All of the fast 2D detectors have a problem with ghosting, which can be eliminated when implementing loops with blank readouts (dead time) in between periods of counting. Latter setup will require a fast shutter or a gated detector.
- The position of the area detector is very important for the final specification of the diffractometer and thus should be determined as soon as possible. The 2D detector will not be mounted on the diffractometer nor a vertical translation system but should have its own horizontal support system for improved stability. Different scenarios for the support system were discussed. The option of a table with air pads was not favored because from operations point of view the realignment of the detector (SDD, tilt and yaw) is rather time consuming. Instead the support table should be positioned on tracks mounted on the hutch floor allowing the detector table to be moved parallel to the beam. The rails need to be short enough so that they do not interfere with the rails of the sample environment table positioned on the floor hutch perpendicular to the beam. Automated detector movement of 200-2000 mm SDD is desirable. Minimum SDD will be dictated by the sample environment. Movement of the detector table parallel/perpendicular to the beam will be very limited, just enough so that the detector can move off center to reach $2\theta = 60$ degrees ($Q = 30$ Å^{-1}) at the minimal distance of 200 mm. A value of $Q = 40$ Å^{-1}cannot be achieved because of the intensity degrading polarization in the horizontal. The detector support should allow the adjustment of the tilt and yaw of the area detector to align the detector as precisely as possible (Eulerian cradle). The final layout of the diffractometer should include all detectors envisioned in the future (as far as this is possible) to avoid collisions and unforeseen overlaps, while maintaining maximum flexibility.

Sample Environments

- Sample environments can be either mounted on the diffractometer or on the support table in front of the diffractometer. Depending on the weight of the sample environment mounted to the diffractometer the footprint of the diffractometer will increase. Thus, a decision on the maximum load needs to be made and the footprint of the diffractometer constrained.

- The size and shape of the table for sample environment can be laid out after the footprint of the diffractometer is fixed. It is suggested to build a table with a footprint of max. 1000 x 1000 mm^2. The height of the table needs to be further discussed because the support for the area detector must protrude it so that one can reach an SDD of 200 mm. The table for the sample environments should move on rails on the hutch floor perpendicular to the beam and should be positioned as close as possible to the circle of the diffractometer to guarantee maximum support.
- The table of the sample environment could make it difficult to enter the hutch if the table is moved all the way outboard. Hence one should consider moving the hutch door further upstream to allow free access to the hutch at all times.
- Space for the sample environments will be 300 x 300 x 300 mm^3 and thus will fit on the proposed 1000 x 1000 mm^2 footprint of the sample environment table.
- General comment: There are no representatives from industry to propose standard sample environments.
- The halogene lamp furnaces can reach very high temperatures (above 2200 ^oC). However, it is difficult to control the temperature in the furnace because the performance of the light bulbs degenerates over time and the Pt/Rh thermocouples melt at 1800-2000 °C. At lower temperatures one can use Pt capillaries that will withstand temperatures up to 1800 °C and that will also work as an internal standard. Alternatively, one could use the very stable $MoSi_2$ furnace that can be controlled with the Pt/Rh or a W/Re thermocouple up to 1800 °C and crosschecked with an internal Pt capillary standard.
- Industry requests a sample environment consisting of a furnace that reaches 1300 °C and imposes a CO gas atmosphere to create reducing conditions. Such a furnace can be built but will be a custom application because the proposed quartz glass chamber for reactive gases developed by the APS can only reach 1000 ^oC.
- There are also requests for lower temperature furnaces that work in conjunction with catalytic reaction chambers. Such a chamber could be combined with the above mentioned CO furnace.
- Sample environments that require levitation of the sample (laser heating for melt studies) are becoming a frequently requested setup (e.g. ID11). However, at this point such a sample environment does not yet seem to be standard and thus should be designed and constructed by specialized users.
- The cold finger cryostat should be replaced by a cryo-streamer since it prevents background and can be operated for several days without icing up when the AC is turned off.
- A robotic sample changer is viewed a high priority after the beamline has been commissioned, especially in conjunction with the cryo-streamer and the hot air blower. Both can operate quasi simultaneously (cryo from above, hot blower from the bottom) with only minor changes in setup. This would allow many communities to process a large numbers of samples in very short time. The suggested development of a mail in service to give other communities and industry access to the facility received critical comments because of the difficulty in processing and interpreting the data without the users being on site.

- Gas supplies for the different sample environments will be located in three dedicated gas cabinets that will be shared between the HRPD and the ECB beamline and are located in sector 2. Gas bottles can no longer be stored in the experimental hutch.

Preparation Labs

- Close proximity of the sample preparation labs to the experimental hutch could be beneficial for some samples but is not an absolute.
- Sealing of capillaries in the sample lab with a Bunsen burner and in the glove box with a glue gun should be standard.
- Fume hood is located in the preparation lab of the Extreme Conditions Beamline and will be operated shared between the two beamlines. PETRA III infrastructure provides also access to chemical labs and clean rooms.
- DESY is working on implementing new policies for the save handling and storing of nano crystalline powders.